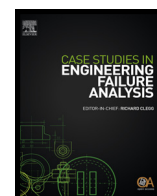


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## Case study

## Failure analysis of edge discoloration of galvanized fuel tank

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## ABSTRACT

A peculiar type of edge discoloration defect on the surface of some galvanized fuel tank was observed, causing significant appearance problems. In the present study, the surface defect was characterized by visual inspection, optical microscopy, scanning electron microscopy and energy dispersive spectroscopic analysis to understand the source and mechanism of the defect. In the visual inspection, these peculiar surface appearances were observed in fuel tank at three distinct locations. The SEM examination exhibited two distinct regions on the surface apart from the normal galvanized surface: (1) galvanized, (2) mixture of galvanized and galvanized texture. The energy dispersive spectroscopic analysis of galvanized region indicated enrichment of Zn and Al whereas in the region of galvanized majorly Zn was observed. Surface texture of galvanized region showed majorly zeta crystals along with skin pass marks; whereas no such zeta crystals were observed in case of galvanized regions. Based on the preliminary results, the following hypothesis was made: Coil processed during galvanizing to galvanized transition. Thickness and width changed to wider and thicker section, which resulted into lower line speed. Due to the lower Al content, lower speed and thicker section combination resulted in formation of partial GA in the coil owing to the internal heat content of the coil. This paper presents the results of the investigation.

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## 1. Introduction

Hot-dip galvanizing is known as the most common technique for protection of steel sheets and structural sections from atmospheric corrosion although it can affect the forming characteristics of steel [1]. Due to the competitive manufacturing of coated sheets, there is an increased interest to produce high quality galvanized steel sheets with minimum defects [2]. Nevertheless, in spite of vast progresses in science and technology of galvanizing process, production of defect free coatings remained a problem, particularly in continuous hot-dip galvanizing. Microstructural defects reduce the formability and corrosion resistance of steel sheet and macroscopic defects damage the surface quality to such extent that may lead to down grading the products in applications with high surface quality requirement, e.g. automobile panel [3,4]. Hot dip galvanizing process usually carried out at a bath temperature of  $460 \pm 5^\circ\text{C}$ , while galvannealing is carried out at  $525 \pm 10^\circ\text{C}$ .

One of the common defects, appeared as dull surface appearance on coated steel sheets, is known as discoloration that produces dull regions on the surface of galvanized coatings. The defect is usually produced due to combination of lower line speed and thickness section [5]. The operational issues usually encountered during typical galvanizing and galvannealing

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process affect the product quality. Characterization of the surface defect using optical and scanning electron microscopy aids in understanding the cause of the defects and helps in designing preventive measures to be taken.

In our present study, the surface defect was characterized by visual inspection, optical microscopy, scanning electron microscopy (SEM) and energy dispersive spectroscopic analysis (EDS) to understand the source and mechanism of the defects.

## 2. Experimental procedure

The fuel tank analyzed in our study was formed from a hot dip galvanized steel sheet of cold rolled IF grade having a thickness of 1.5 mm. The composition in wt.% of the steel sheet was C-0.0024, Mn-0.06, S-0.006, P-0.013, Si-0.005, Al-0.058, Cu-0.005, Cr-0.015, Ni-0.016, Mo-0.001, V-0.001, Nb-0.001, and Ti-0.063. The coating weight was about 50–70 g/m<sup>2</sup> on each side. A sample was cut from the defect region of the fuel tank for investigation purpose. Visual examination was carried out on the defect sample of galvanized sheet component. The defects in as-received galvanized sheet were also observed in an optical microscope.

SEM examination was carried out on the as-received galvanized fuel tank using Field Emission Gun Scanning Electron Microscope (FEG-SEM). In order to identify the chemical composition of the various regions observed in SEM, an energy dispersive spectroscopy (EDS) analysis was carried out. In order to identify the source of defect, surface topographic SEM examination was carried out. The elemental profile of various elements over the defect region was obtained at  $5 \times 10^{-8}$  A probe current and 15 keV accelerating voltage. Before SEM analysis, sample from bright and dull region was cleaned using ultrasonic cleaner ELMA S30H. For microstructural analysis samples were individually mounted in electrically conductive copper-containing resin and polished by conventional metallographic techniques. The polished samples were etched with 3% nital solution (3 ml HNO<sub>3</sub> in 97 ml ethyl alcohol) for analysis of microstructure and studied under light optical microscope (LOM).

## 3. Experimental results

### 3.1. Visual inspection

The photograph of the fuel tank made from galvanized steel sheet consisting of peculiar shade difference is shown in Fig. 1. These defects are readily observed by naked eye. From the photograph it can be seen that the top surface sheet have bright appearance whereas bottom surface of sheet have dull appearance as shown by the arrows. At some location, grain structure like appearance is also observed.

### 3.2. Optical microscopy

Through thickness micro specimens prepared from the fuel tank for coating evaluation at top and bottom surface. These micro specimens are studied under light optical microscopy at different magnification. The optical micrograph of top surface

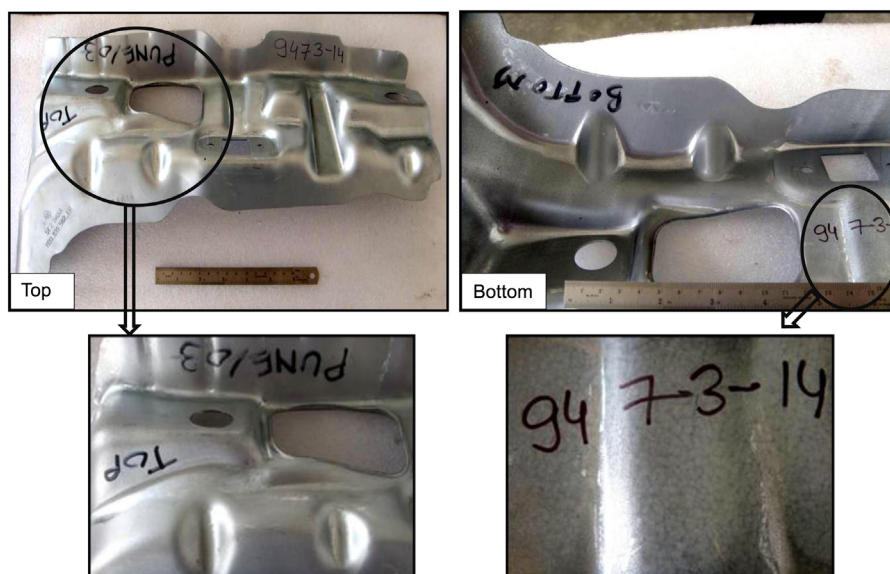


Fig. 1. Photograph of the galvanized fuel tank consisting of peculiar shade difference problem.

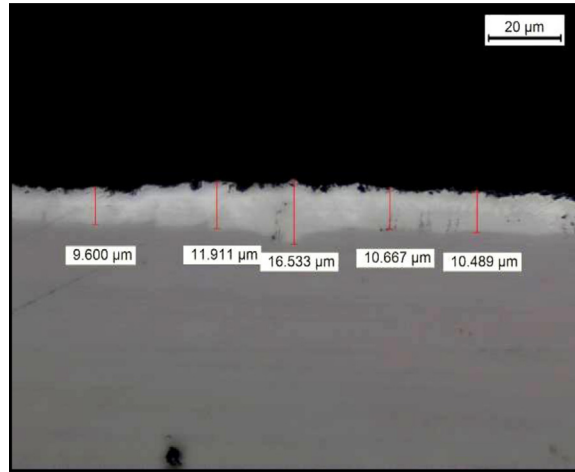


Fig. 2. Optical microphotograph of the top surface of Galvanized fuel tank.

is shown in Fig. 2; whereas bottom surface is shown in Fig. 3. Uniform coating is observed on top surface with an average thickness of 11.84  $\mu\text{m}$ ; whereas bottom coating appeared as brittle in nature. Average coating thickness at bottom surface was 10.58  $\mu\text{m}$ . Coating thickness of top surface having bright appearance is more than the coating of bottom surface.

### 3.3. SEM examination

The SEM microphotograph from the defect region, having dull appearance on the galvanized fuel tank is shown in Fig. 4a. Fig. 4a shows two distinct regions, viz. Region A is the zeta crystals, region B is the skin pass marks. Fig. 4b is the high magnification SEM micrograph of region A and B in Fig. 4a. From Fig. 4b we can see that the zeta crystals are clearly visible indicating Galvannealing take place at this region. EDS analysis was carried out on Galvannealed region showing the concentration in wt.% of Zn, Fe and Al in these regions as mentioned in Table 1.

The SEM microphotograph from the defect region, having grain structure like appearance on the galvanized fuel tank is shown in Fig. 5a. Fig. 5a shows two distinct regions, viz. Region A is the partially zeta crystals, region B is the skin pass marks. Fig. 5b is the high magnification SEM micrograph of region A and B in Fig. 5a. From Fig. 5b we can see that the zeta crystals are partially appeared over the surface indicating transition zone of Galvannealing and Galvanizing at this region. EDS analysis was carried out at this transition region showing the concentration in wt.% of Zn, Fe and Al in these regions as mentioned in Table 2.

The SEM microphotograph from the non-defect region having bright appearance shows Galvanized surface appearance along with skin pass mark as shown in Fig. 6a. Fig. 6b is the high magnification SEM microphotograph of Galvanized surface. EDS analysis was carried out at this bright region showing the concentration in wt.% of Zn, Fe and Al in these regions as mentioned in Table 3.

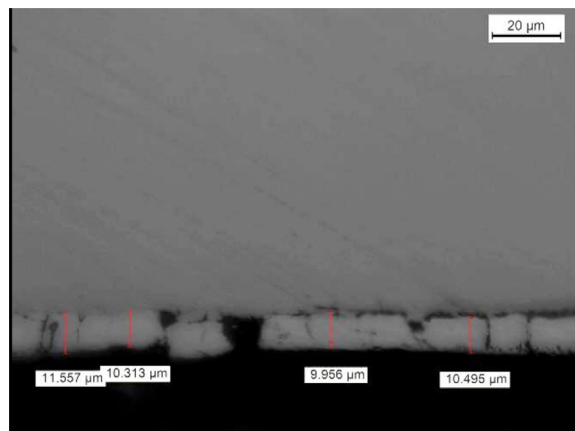
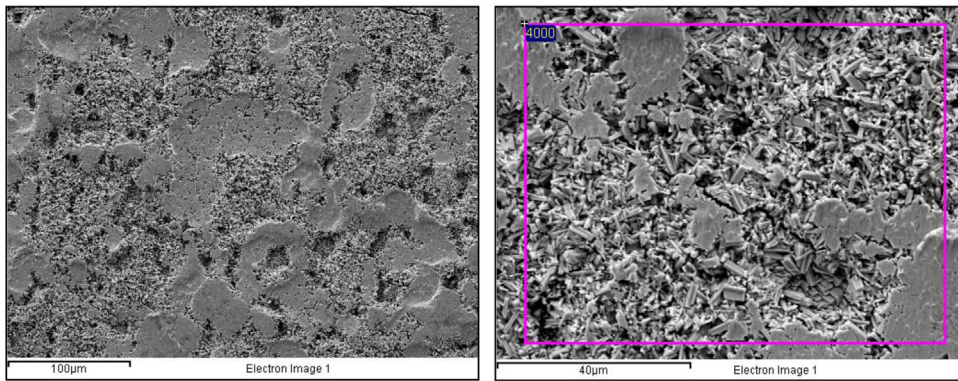


Fig. 3. Optical microphotograph of the bottom surface of Galvanized fuel tank.



**Fig. 4.** (a) SEM microphotograph of Galvannealed surface at 1000 $\times$  magnification. (b) High magnification (4000 $\times$ ) SEM microphotograph of Galvannealed surface.

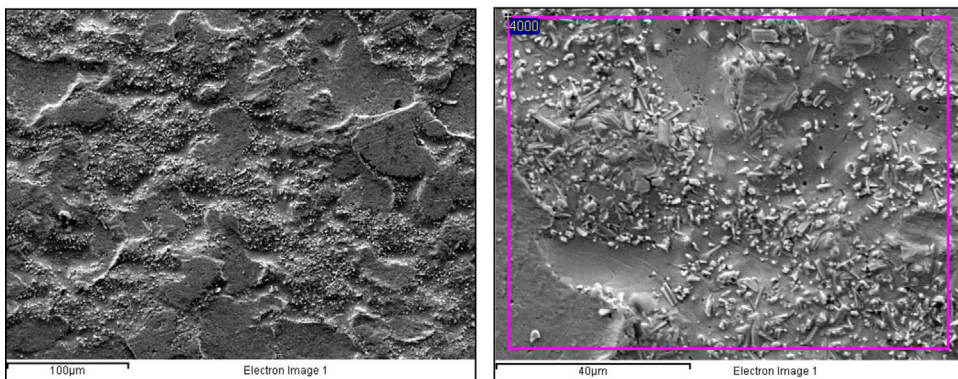
**Table 1**  
EDS analysis of dull surface (area analysis).

| Element | Weight% | Atomic% |
|---------|---------|---------|
| O K     | 2.79    | 10.28   |
| Al K    | 0.61    | 1.34    |
| Fe K    | 8.33    | 8.79    |
| Zn L    | 88.27   | 79.59   |

#### 4. Discussion

Visual observation of the defective fuel tank confirms that there are three regions bright, dull and grain structure like appearance on the surface. This grain structure like appearance is result of transition of galvanizing and galvannealing process. SEM microphotographs and EDS analysis shows the presence of zeta crystals and high amount of Fe in dull surface, it confirms that galvannealing take place at bottom surface. Amount of Fe in top surface is low which resulted into bright surface. Optical microscopy study confirms the presence of brittle coating layer at bottom surface.

Based on the preliminary results, the following hypothesis was made: The dull appearance of the bottom surface is due to the partial Galvannealing (GA) formation. The metallographic analysis shows the partial GA formation on both bottom and top surfaces though the extent of GA formation is greater on the bottom surface due to which the appearance is darker as compare to top surface. The complaint coil was processed during galvanizing to galvannealing transition when the zinc bath aluminium run down was going on. There was thickness and width transition and the coil was processed at a lower line speed on account of its thicker section.



**Fig. 5.** (a) SEM Microphotograph of transition region of Galvannealing and Galvanizing at 1000 $\times$  magnification. (b) High magnification (4000 $\times$ ) SEM microphotograph of transition region.



**Table 2**

EDS analysis of Grain structure like surface appearance (area analysis).

| Element | Weight% | Atomic% |
|---------|---------|---------|
| O K     | 2.73    | 10.16   |
| Al K    | 0.53    | 1.16    |
| Fe K    | 3.27    | 3.49    |
| Zn L    | 93.48   | 85.20   |

**Table 3**

EDS analysis of bright surface (area analysis).

| Element | Weight% | Atomic% |
|---------|---------|---------|
| O K     | 3.61    | 13.13   |
| Al K    | 0.50    | 1.08    |
| Fe K    | 2.29    | 2.39    |
| Zn K    | 93.61   | 83.41   |

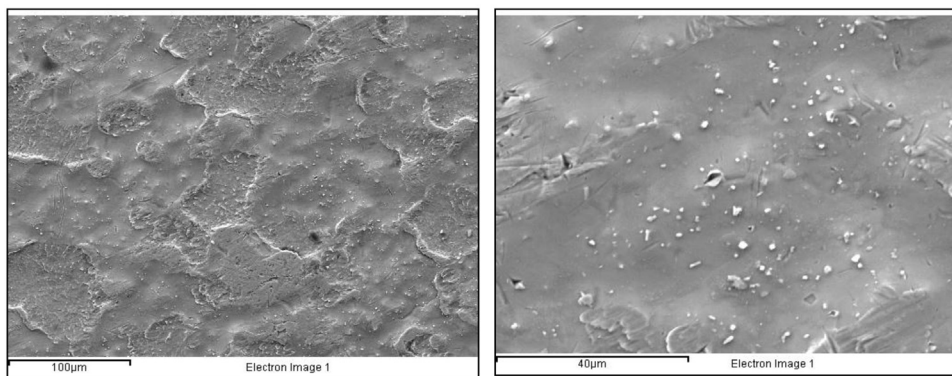


Fig. 6. (a) SEM microphotograph of Galvanized surface at 1000 $\times$  magnification. (b) High magnification (4000 $\times$ ) SEM microphotograph of Galvanized surface.

## 5. Conclusion

Due to lower bath aluminium content ( $\sim 0.13\%$  as compared to  $0.185\%$  regular during galvanizing process), lower speed and thicker section combination resulted in formation of partial Galvannealed surface in the coil owing to the internal heat content of the coil. It is recommended that coils for automotive galvanizing grade are not to be scheduled during unstable zinc condition (like run down).

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